A NUCLEATION EXPERIMENT IN LOW GRAVITY CONDITIONS: MONITORING AND COLLECTION OF Mg AND Zn PARTICLES

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ABSTRACT

An experiment on the nucleation and growth of solid particles from the condensation of vapor in low gravity conditions was performed onboard NASA's KC-135 Microgravity Research Aircraft. In addition to the systems to monitor relevant physical parameters, two systems have been developed and used to observe the growth process of the particles and to extract these particles for further microscopic analyses. A dynamic light scattering system is used to measure the *in situ* mean particle size while a second independent system allows the collection of the nucleated particles directly from the condensation front. These samples have been analyzed in the lab using Scanning Electron Microscopy (SEM). Mean particle sizes are approximately 300 nm and 150 nm for Mg and Zn particles, respectively. Evidence for particle-particle coagulation of Mg into "fractal" like clusters is seen while Zn shows no evidence for any coagulation over the conditions explored.

INTRODUCTION

The condensation behavior of high temperature vapors is important in such diverse fields as vapor deposition, stratospheric chemistry, and the formation of cosmic dust. Cosmic dust, formed in the atmospheres of stars, can have a major influence on the thermal balance of stars, serve as reactive sites for circumstellar and interstellar chemistry, and may dominate the optical opacity of interstellar regions. Significant progress has been made in recent years in identifying the types of materials that compose cosmic dust. In all astrophysical regions hydrogen is by far the most abundant element, however, the relative concentration of condensable elements determines the chemistry of the dust formed. In areas where the oxygen to carbon ratio exceeds unity grains containing (Mg, Fe, Al, Ca, and Na) are believed to occur as amorphous silicate grains. Whereas in stellar regions of high C/O ratio, carbon and metal carbide grains are expected to condense (Bussoletti and Colangeli, 1990). While the major classes of cosmic grain materials have been identified, details of the size and shape of grains and the mechanisms of grain formation and growth are still unknown.

Laboratory synthesis of cosmic dust analogs have been quite successful in determining the possible materials that match the observed astronomical spectral signatures (Hallenbeck *et al.*, 1998). These analog grains have been produced via high temperature homogeneous nucleation by the gas evaporation method (Nuth and Donn, 1982). Unfortunately, in order to properly simulate the formation of cosmic dust analogs in the laboratory, and to study their growth, closer control is needed on the conditions in which grains are formed; and the laboratory conditions need to more closely mimic environments where grain formation occurs. Both of these requirements can be met by performing gas evaporation studies of possible candidate materials under low or microgravity conditions. As a result we have designed and built an apparatus that can allow *in situ* particle size determination and additionally allow for extraction of individual particle samples for later microscopic analysis. In order to produce the required low gravity conditions, the entire apparatus was designed to fly aboard NASA's KC-135 Microgravity Research

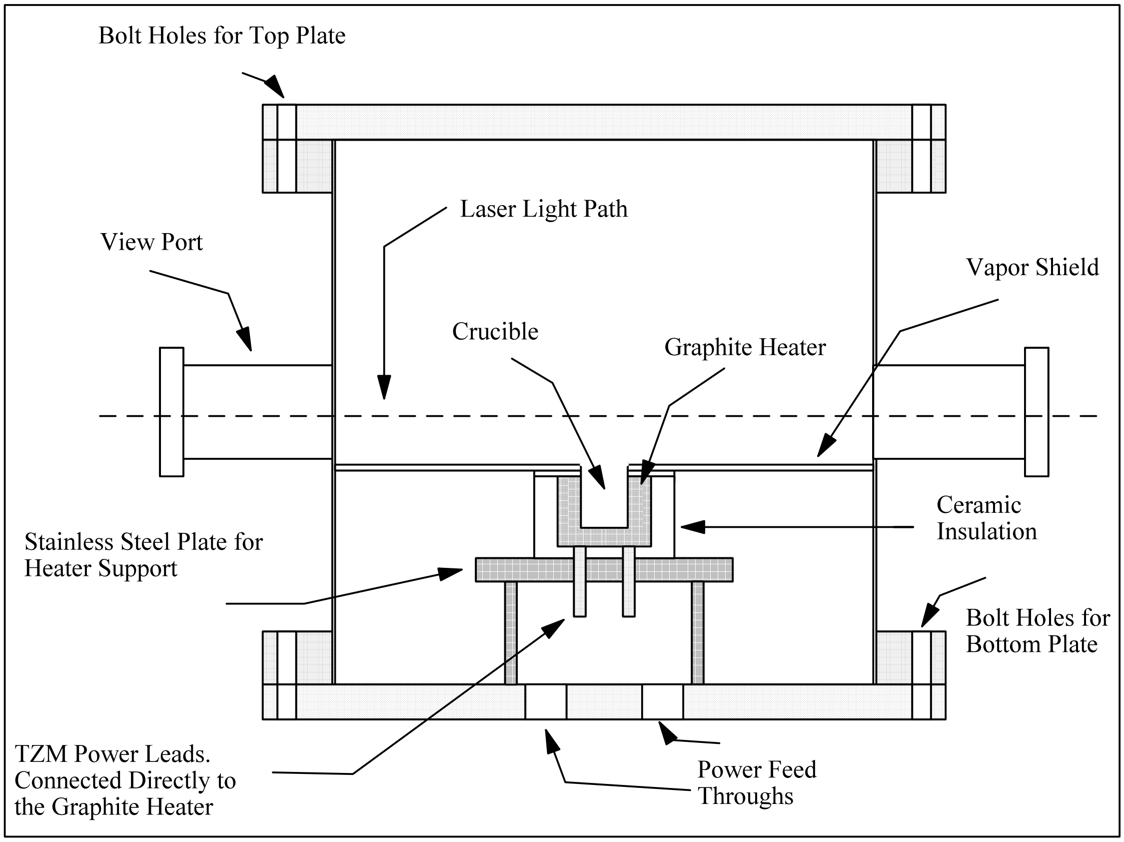

Fig. 1. Cross-section of experimental chamber

Aircraft. This aircraft flies in a series of parabolic arcs and can produce up to 23 seconds of low gravity conditions (10^{-3} g). While the periods of low gravity produced by the KC-135 allow only the initial stages of particle coagulation to be studied, they provide an excellent environment to validate ideas and equipment for further studies aboard the space shuttle and space station.

EXPERIMENTAL APPARATUS

To study the initial stages of particle growth and coagulation of the resulting grains we have designed and built the chamber shown in Figure 1. It consists of a cylindrical vacuum chamber separated into two main sections. The upper section is devoid of all extraneous surfaces that could initiate heterogeneous nucleation. In addition, there are eight concentric ports spaced evenly around the upper section that provide access for the light scattering system, particle extraction system, and vacuum pumping, etc. The lower section houses the graphite heater and associated feedthroughs for electrical and thermocouple wiring. A total of 20 thermocouples are placed strategically within the heater region and embedded into the vapor shield and upper section walls for recording of boundary and vaporization temperatures. The pressure, acceleration level, temperatures throughout the chamber and the data from the light scattering system are recorded using a computerized data acquisition system.

Photon Correlation Spectroscopy (PCS) is employed with the light scattering system using all solid state components. The laser is a solid state diode producing 10 mW of power which is launched into single mode fiber optics. The fiber optics route the radiation to the launching point within one of the eight concentric ports on the upper section of the chamber. A second set of fiber optics runs from another port orthogonal to the point of scattering. This “captured” light is then routed down to an EG&G single photon counting module (SPCM). The output of the SPCM is connected to an in-house designed correlator which takes in the photon counts and creates an autocorrelation function. The final particle size distribution is then found by inverting the autocorrelation function using a Maximum Entropy Method (MEM) formalism and assuming that the resulting particles are spherical in shape. The actual numerical methods are based on the work of (Michael, 1998) and (Nyeo and Chu, 1989).

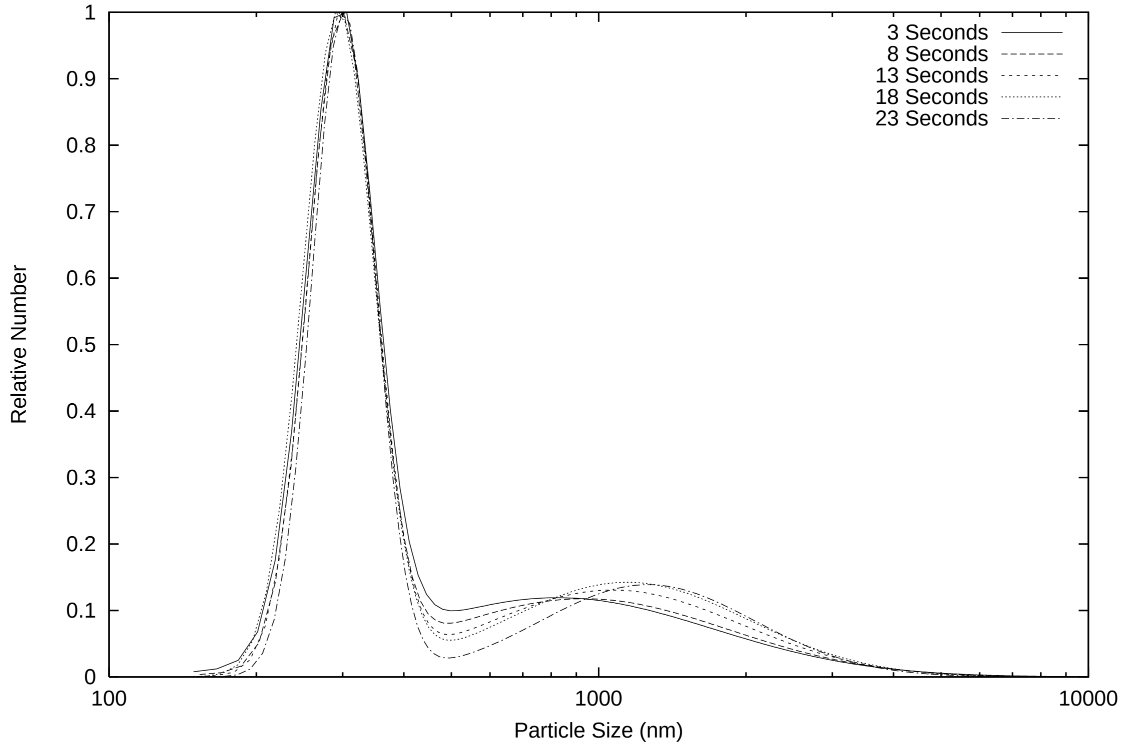

Fig. 2. Magnesium particle size at 5 second intervals during 23 seconds of microgravity

RESULTS

Magnesium

Figure 2 shows a series of particle size distributions for magnesium during a single microgravity parabola. The first peak at around 300 nm is representative of the mean size that we see throughout the pressure (20 - 60 torr) and temperature (900 - 1200 K, point of vaporization) ranges explored. Temperature does not seem to play a significant part in determining particle size for magnesium, pressure changes on the other hand can have significant effects on the breadth of the size distribution. Decreases as small as 10 torr can increase the particle size distribution from 200 - 400 nm out to as wide as 100 - 800 nm. The mean particle size of 300 nm is confirmed by Scanning Electron Microscopy (SEM) analysis and shows that particles are spherical in nature. Evidence for smaller particles (50 nm) is seen in the SEM analysis, but these particles are rapidly consumed into larger 300 - 500 nm single particles and a few agglomerates. Unfortunately, initial individual particle growth occurred mainly below the position of our light scattering system and no evidence was seen of simple growth from the vapor.

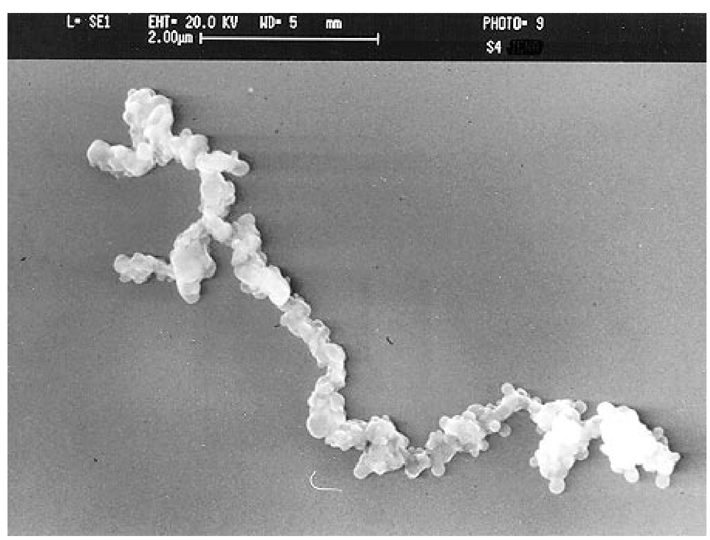

Fig. 3. Micrograph of coagulated magnesium particles showing the “fractal” like characteristics

Evidence for particle coagulation is seen in both the light scattering, second peak of Figure 2, and the SEM. The SEM analysis shows that the coagulated particles form large open “fractal” like clusters, Figure 3. This type of coagulation is exactly the type predicted by several theories on particle coagulation within environments that are mainly diffusion limited, as might be expected in large interstellar dust clouds. Unfortunately, the light scattering system only

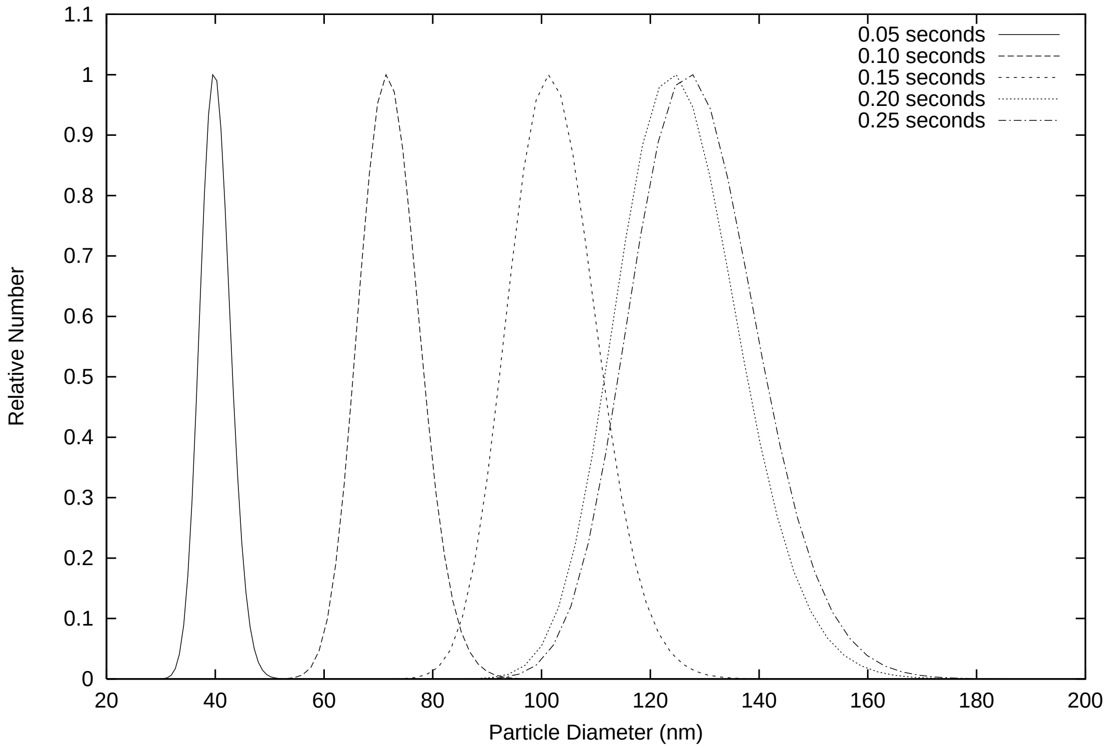

Fig. 4. Zinc particle size at 0.05 second intervals during growth phase in microgravity

showed limited evidence for coagulation, with such evidence limited to the size range between about 500 - 1500 nm diameter particles. One possible reason for our limited light scattering evidence for coagulation is that our present system has limited ability to resolve peaks with a size ratio less than 3. We hope to correct this problem by adding additional detectors to our system enabling a multiple angle setup instead of the current single angle setup.

Zinc

While magnesium showed evidence for coagulation, even in the relatively short period of microgravity, zinc did not display any evidence for coagulation. Single individual particles with mean diameters around 150 nm were the norm for pressure and vapor source temperatures in the ranges 20 - 60 torr and 700 - 1200 K, respectively. Whereas magnesium showed changes in the particle size distribution due to pressure effects, zinc exhibited very little change in either mean size or distribution width over the parameters explored. Light scattering analysis consistently underestimated the mean particle size by approximately 20 - 40 nm. This is most likely due to the fact that the zinc particles were not spherical, but instead were "bow tie" shaped. This difference in size resulted from using an assumption of spherical particles within the autocorrelation inversion method and in the final calculation of the particle size with a Stokes-Einstein relationship.

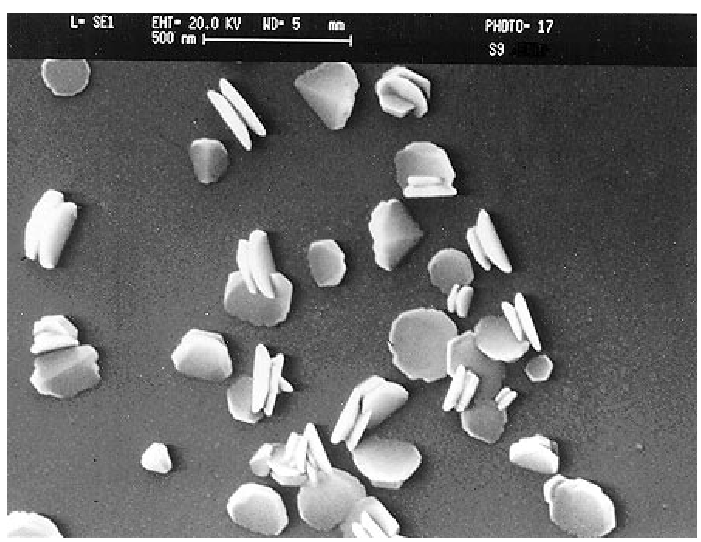

Fig. 5. Micrograph of zinc particles showing the characteristic "bow tie" structure

Figure 4 displays the results for zinc particle growth during 0.25 seconds after nucleation had occurred. Particle growth occurred almost exclusively during a short period (0.40 seconds) immediately after nucleation indicating growth was from the vapor and that growth rapidly scavenged up the available zinc vapor leading to very little further growth. SEM analysis (Figure 5) showed that particles were the familiar “bow tie” shapes seen by other researchers (Eversole and Broida, 1974), further reinforcing the idea that zinc particle shape is influenced more by its size than by the initial physical properties of the vapor.

SUMMARY

Magnesium particles show evidence that coagulation can occur in the relatively short periods of microgravity available with the KC-135 Research Aircraft. The coagulated particles resemble open “fractal” like clusters such as those predicted by several theories on particle coagulation in diffusion limited environments. Mean particle size is 300 nm and 150 nm for magnesium and zinc, respectively, with magnesium particles spherical in shape and zinc particles reminiscent of “bow ties”. Temperature and pressure seem to play little to no role in the size or shape of zinc, but in the case of magnesium pressure can have a drastic effect on the width of the size distribution.

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